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An Undergraduate Honors College Thesis

in the

College of Engineering
University of Arkansas
Fayetteville, AR

by

This thesis is approved.

Thesis Advisor:

A large, stylized handwritten signature in black ink, likely belonging to the Thesis Advisor, written over the signature line.

Thesis Committee:

Ultrasonic HogUAnauts

WERC Task 2: Separation of Oil from Water

Over the course of the Spring 2015 semester I was a member of one of the University of Arkansas teams that won 1st place competing in the 2015 WERC Design Competition. Participating in Task 2, my team, Ultrasonic HogUAnauts, were charged with the development of a process that could break an oil-in-water emulsion separating it into pure-water and pure-oil streams. As the Research Coordinator for the team, my responsibilities involved the organization of the team's technology research efforts and the creation of an experimental program to develop a bench-scale model. Furthermore, I was involved in the making of the team's poster and pamphlets that were used during the bench-scale presentation, and I was a part of the four-person team responsible for presenting the HogUAnauts' design to a panel of judges at the competition in Las Cruces, NM.

The evaluation criteria for Task 2 of the 2015 WERC Competition had two major requirements for the competing teams. First, the teams were required to demonstrate their ability to create an oil-in-brine emulsion via sonication. Then, they would be judged on their ability to break the emulsion and separate it into pure-oil and pure-brine. In order to fully address both requirements, the HogUAnauts' first order of business was to research sonication techniques and to explore different technologies used to remove oil from oil-in-water emulsions. Using a WERC report from the 2013 FracHogs team as a starting point, I delegated research topics to each group member. For this preliminary research stage, I was heavily involved in researching coalescers as a means to separate oil from oil-in-water emulsions.

Once enough research had been conducted, the HogUAnauts arrived at the conclusion that the most appropriate method of sonication was to use a dual-frequency sonicator already owned by the Chemical Engineering department and that the technologies most likely to yield a simple, environmentally friendly and cost-effective way of breaking and separating the emulsion were ultrafiltration and coalescing. In order to determine how best to adapt these technologies into a design that would meet the requirements established in Task 2, it was my responsibility as the research coordinator to plan an experimental program for the team to follow.

The first item in the experimental program was to determine the dual-frequency sonicator's range of emulsion-making capabilities and determining whether it would in fact be the best method of achieving an emulsion that measured up to competition standards. This involved the testing of how different frequencies, sonication times and recirculation methods affected the tightness of the resulting emulsion. In order to quantify the quality of the sonicated emulsions, a microscope was used to analyze emulsion samples measuring the size of the resulting oil droplets dispersed throughout the brine water.

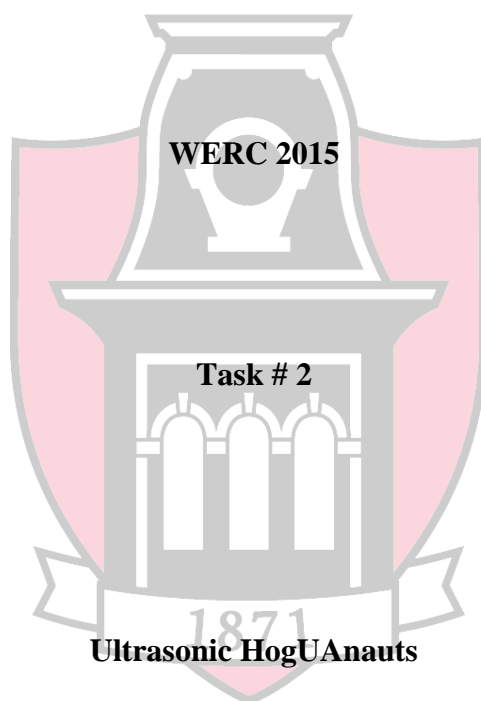
Second, the HogUAnauts called on the expertise of Dr. Robert Cross in order to guide the research that would lead to the ultrafiltration scheme that would be used for the bench scale apparatus. Using hollow-fiber ultrafiltration membrane cartridges provided to the department by Koch Industries, various degrees of volume reduction were tested. By taking freshly made emulsions and filtering out pure brine, the oil concentration in the emulsion was increased in order to achieve concentrations that would be optimal for separation in a coalescer. For this part of the experimental program, cartridge cleaning methods were also investigated to counteract the effects of membrane fouling.

Finally, the last step in the experimental program was to develop a coalescer capable of separating the emulsified oil and separating it into a pure-oil stream. Several coalescer designs were tested beginning with a simple syringe attached to a ¼” copper tube stuffed with a fiber bed and evolving into a combination of a gravity separator tank and a coalescing chamber with a packed bed of polypropylene fibers.

As the research coordinator, I was heavily involved not only in the planning of the experimental program, but also in its execution having a role in the majority of the experimental tests conducted by the team. Throughout the development of the HogUAnauts’ process, I was involved in meetings with experts like Dr. Cross and Dan Trantham and in reaching out to companies such as Alanco Technologies, Inc. and Apache Corp. In order to replicate the conditions that the HogUAnauts would encounter at the competition, I was also in charge of securing brine from the Brackish Groundwater National Desalination and Research Facility.

Once the HogUAnauts bench-scale had been developed and the process had been scaled up to treat the 100 gpm of oil-in-brine emulsion specified in Task 2, I was heavily involved with the creation of the poster and the pamphlets that would be used during the bench-scale presentation at the competition in New Mexico. As the research coordinator, I was chosen as one of the four people responsible of presenting the HogUAnauts design to a panel of judges and was tasked with illustrating to them the experimental process that led to our final design.

SEPARATION OF OIL FROM WATER



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SEPARATION OF OIL FROM WATER

Task # 2

Ultrasonic HogUAnauts

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EXECUTIVE SUMMARY

In the past 40 years, a variety of enhanced oil recovery (EOR) methods have been developed and applied to mature, mostly depleted, and shale formation oil reservoirs. Chemical and sonic stimulation are two enhanced oil recovery methods in which emulsions are created either as a primary or secondary effect. The resulting viscosity of the oil in water emulsion is considerably lower than that of dry crude, thus increasing recovery from pay zones.

During chemical enhanced oil recovery, caustic or surfactants are injected into oil reservoirs, which results in the creation of stable oil-water emulsions. The emulsions from chemical enhanced oil recovery floods can be very stable, and as such, traditional demulsifiers are often not effective.

Sonic stimulation is performed by the insertion of a piezoelectric (or other type) transducer into a well and exposing a pay zone to a set of frequencies for a period of time. This new technology is still being researched; however, results have been promising. Research conducted at Pennsylvania State University has demonstrated stripper well production increases of approximately 30% after *in situ* well sonication.

These stimulations, along with seismic activity, can generate significant volumes of emulsion that need to be broken in order to produce commercially dry oil, and meet clean water requirements that oil producers seek to achieve. A typical production specification is an oil phase containing no more than 0.3 - 0.5% water by volume and an aqueous phase containing no more than 200 ppm oil, preferably < 100 ppm.

When considering alternatives for oil-in-water demulsification, there are various options that can be considered. The use of pH manipulation was investigated, however the addition of harsh chemicals is not ideal and only mildly effective. Multiple effect evaporation will produce potable water; however, the energy and capital costs are high. Another option is to use centrifugal separation, which is capable of achieving high degrees of separation but it is energy and capital intensive. Coalescence, which was investigated, is particularly attractive because of its simplicity and efficacy. Ultrafiltration, also investigated, is highly effective at producing oil free brackish water but cannot produce a pure oil stream. Due to the low concentration (~ 200 ppm) of oil in the feed, ultrafiltration was paired with coalescence to produce a brine free of dispersed phase oil and a marketable oil stream.

The WERC task statement specifies a high degree of removal of oil from the brackish water stream. The full-scale process will be robust, remove all of the dispersed oil from a 100 gpm feed stream, and produce oil with low water content, preferably marketable.

Laboratory work produced brackish water filtrate free of any dispersed oil and produced an oil phase substantially free of water, deemed marketable. Additionally sonication was used very successfully to produce an oil in water emulsion with an average droplet size < 6.0 microns.

The full-scale UF/Coalescence process was designed to be highly mobile to satisfy the transient nature of the fracking industry. The capital cost for this process to separate all the entrained oil from the oil in water dispersion is \$250,000 and the operating costs are less than \$20,000/year, excluding any additional operating labor. At 8,000 hrs/year of operation, 4 operators will be required, incurring an added annual operating cost of \$200,000 to \$250,000.

INTRODUCTION

The recent boom of hydraulic fracturing, or “fracking”, within the US has led to an increase in the production of flowback water. Flowback water from hydraulically fractured shale gas and oil wells typically contains emulsified oil, dissolved solids, and other contaminants. These contaminants render the water unsafe for the environment and potentially harmful to water treatment equipment.¹

The primary purpose of Task # 2 is to develop a process for separating oil from water such that the water can be reused in a plant or process. Several types of processing equipment are commonly used to achieve this separation including gravity separators, centrifuges, ultrafiltration units, and coalescers. These can achieve differing degrees of separation but vary widely in cost of implementation and efficacy for emulsions of various stabilities.²

A process with high separation such that the oil and water can be removed separately using decantation is desirable.³ The separated oil will typically be of sufficient quality to be marketed² and the water can be reused in fracking operations, disposed of via deep well injection, or further treated.

TASK PARAMETERS

The design premises specified for this task are to:

1. Demonstrate applicable sonication technology for dispersing vegetable oil in brackish water.
2. Remove the emulsified oil from the brackish water sample.
3. Design a commercial scale, cost effective water treatment system that handles 100 gpm of oil contaminated brackish water.
4. Maximize the degree of separation and purity of recovered oil and water.
5. Minimize energy use.
6. Maximize ease of operation, reliability, and safety.
7. Process 10 gallons of brackish water with a concentration of 200 mg/L of oil in a bench scale apparatus.

TECHNOLOGIES CONSIDERED

Sonication

Sonication is the application of acoustic or ultrasonic pressure waves to agitate a sample. Sonication has a wide variety of applications ranging from emulsification to ultrasonic cleaning of machine tools. Direct, indirect, and dual frequency sonication were the methods of emulsification considered for this task.

Direct sonication is the most common method of sonication and uses a submerged probe to directly agitate a liquid sample. However, this technique is limited to producing small volumes of emulsion.

Indirect sonication is similar to direct sonication in that it uses a probe or horn submerged in a liquid bath. The sonic agitation is then transmitted to anything submerged in the bath. This is suitable for multiple simultaneous sonications but it is still generally limited to very small sample volumes.

Dual frequency sonication, selected for this process, makes use of multiple transducers to generate two resonant frequencies that interact to form a third “beat” frequency. This results in greatly improved distribution of acoustic energy throughout the sonication vessel. This allows for construction of much larger vessels and continuous flow processing while still achieving thorough agitation.⁴

Oil/Water Separation

There are several water treatment technologies used in industrial systems to remove emulsified oil from water. The oil properties, dispersion characteristics, and other components in

the mixture effect emulsion stability. The technologies that were considered in this study are as follows: pH manipulation, multiple-effect evaporation, centrifugal separation, ultrafiltration, and coalescence.

pH manipulation is a process in which the acidity of an emulsion is elevated. As the pH decreases, the oil droplets' individual charge, and thus mutual repulsion, is diminished. This allows for easier agglomeration and coalescence. For the emulsions produced in this study, the pH was varied from 7.2 to < 1.0 . The effect on emulsion stability was not significant and any water treated with this method would have to be chemically neutralized for downstream use thus greatly increasing both complexity and cost of processing.^{5,6}

Multiple effect evaporation is a continuous process in which water is evaporated in a series of stages with each subsequent stage operating at a lower pressure than the preceding. The reduction of pressure results in a lowering of the boiling point such that vapor from an upstream stage can be used to vaporize water in the lower pressure downstream stage. Once the cascade effect is established, only the first stage requires input of external heat.⁷ Except where volatile oil is concerned, multiple effect evaporation will produce potable water. However, the concentration of salts from stage-to-stage detracts from the boiling point depression effect and also results in accumulation of previously dissolved solids in the oil. This method would have high complexity, capital cost, and energy cost.

Centrifugal separation exerts forces of up to many thousand times that exerted by gravity on a sample. Density differences between phases cause dispersed phase droplets to move towards a coalescing interface between the oil and brackish water. In a solid bowl centrifuge, both phases flow from the separator over weirs, which are positioned to keep the interface well within the bowl. The separated liquids exit through different outlets. Industrial centrifuges achieve a high degree of separation; however, they are large, complex pieces of equipment that require a great deal of energy to operate.⁸

Ultrafiltration (UF) is a membrane separation process in which applied pressure forces a liquid through a porous membrane. The feed is separated into a filtrate stream that permeates the membrane and a retentate stream that does not permeate the membrane. UF is a proven water treatment method that is effective for removing microorganisms, suspended solids, and other solutes and dispersions of high molecular weight. UF is ideally suited to remove dispersed phase oil droplets from brackish water provided the oil does not selectively wet the membrane. The

UF membrane will not separate effectively if the concentration of oil becomes high enough that the emulsion phases invert. While this method would produce essentially oil free water, it will not sufficiently reduce the amount of water left in the oil phase.⁹

Coalescing is a method of reducing residence time in a settling or gravity separation vessel by passing the emulsion through an oleophilic bed of fibers. The oil droplets in the mixture selectively adhere to the surface of the coalescing medium where they agglomerate and coalesce to form larger droplets. These droplets become large enough to be sheared from the fibers and rise to the liquid surface. The efficacy of the bed as a coalescing medium must be determined experimentally because the phenomena causing the coalescence are too complex to predict *a priori*. The efficiency of this process is dependent on the shape and surface area of the oleophilic material, liquid and oil density, and the bulk liquid velocity.¹⁰ This method is operationally simple, low cost, and low maintenance. It also achieves a commercially acceptable level of separation of water from the oil phase.

A combination of UF and coalescence was selected for the completion of Task # 2. This hybrid process was selected due to UF's ability to produce a high purity brine stream and coalescence's ability to produce a relatively dry oil stream. The emulsion exiting the coalescing stage can be recycled to the UF unit such that the only outlet streams are clean brine from the UF unit and oil from the coalescing unit.

DESIGN THEORY

The test emulsion generated for this task has an oil concentration of 200 mg/L and a number average droplet size of < 10 microns. This concentration is low enough that the coalescer alone is not sufficient to break the emulsion in a timely manner. For this reason, the oil is concentrated via UF before being sent to the coalescer.

The primary purpose of the UF unit is to produce clean brackish water. Its secondary purpose is to reduce the total volume of water in the mixture and increase the concentration of oil sent to the coalescer. Using UF at the specified starting concentration of 200 ppm oil, > 90% of the total volume can be removed as clean filtrate without a phase inversion occurring in the retentate. Thus, the required volume of the coalescing vessel has also been reduced by > 90%.

Flow rate is a critical part of the coalescing process. The flow rate needs to be sufficiently high to shear the coalesced droplets from the fibers and the flow rate needs to be sufficiently low so that the droplets will adhere to the fibers and coalesce.

BENCH SCALE APPARATUS

Sonication

Equipment

The dual-frequency sonicator used was manufactured by The Lewis Corporation and its Model Number is NAP-1608-TC. The device has two transducers attached to diaphragm plates on either side of the sonication chamber. One transducer operates at 20 kHz and the other at 16 kHz, producing a beat frequency of 4 kHz. A Little Giant Pump Co., Model Number 71620871 centrifugal pump circulated the oil/brine mixture through the sonication chamber. In 1 hour of sonication emulsions were produced with a number average drop size of < 6 microns. The apparatus is shown in Figure 1.

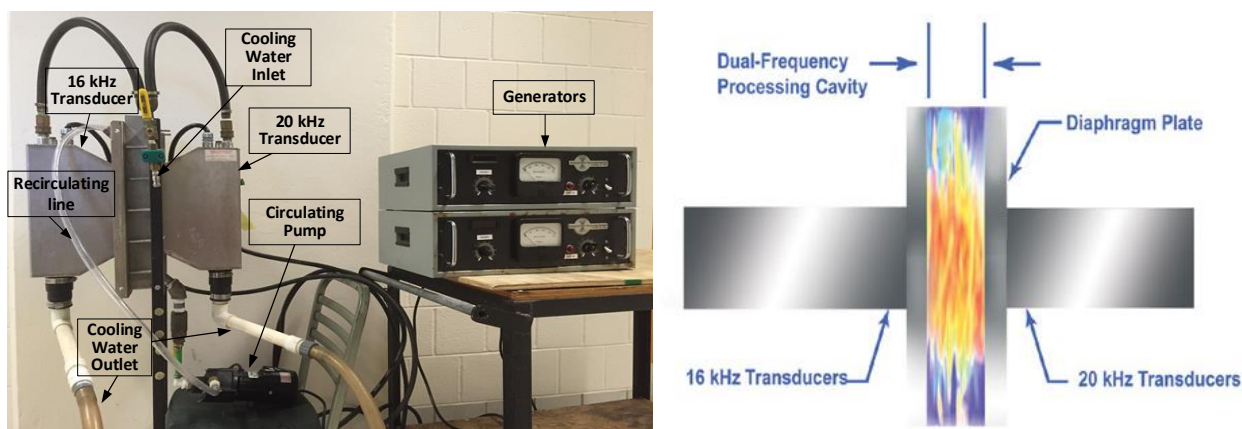


Figure 1. (Left) A Photograph of the Sonicator. (Right) A Diagram of the Distribution of Sonic Agitation.

Operation

1. The processing chamber was filled with 0.74 gallons (2.8 L) of brackish water via a funnel inserted into the threaded hole at the top of the processing chamber.
2. Cooling water was connected and flowed through the heat exchangers in the transducer housings at 1 gpm.
3. Power was supplied to the transducers.

4. The centrifugal pump provided mixing by recirculating the mixture through the sonication chamber at 5 gpm.
5. 6 mL of soybean cooking oil were added quickly from a syringe through a 3" long hypodermic needle.
6. Sonication was applied for about 1 hour, producing a stable emulsion.

Separation of the Oil from the Water

This apparatus consisted of two separate units, a UF unit and a coalescing unit. A process flow diagram of the apparatus is shown in Figure 2.

Equipment: UF unit

1. A 5 L HDPE feed container
2. A 2 gpm Dayton piston pump which was driven by a DC variable speed motor
3. 20' of 1/4" Silicone tubing
4. A 0-30 psi pressure gauge for measuring the inlet pressure of the UF module
5. A Koch 1" HF 1.0-43-F UF laboratory unit with 1 ft² of filtration area
6. A 0-15 psi pressure gauge for measuring the outlet pressure of the UF module
7. A 1/4" ball valve in the outlet line downstream of the outlet pressure gauge which allows the outlet pressure of the module to be controlled at 15 psi

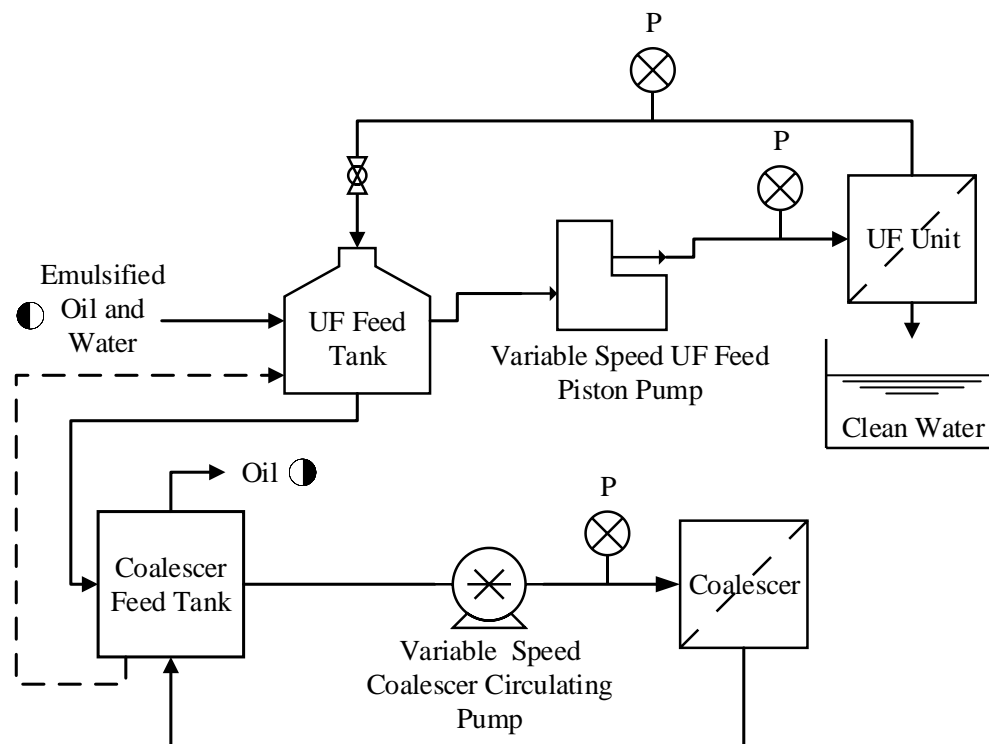


Figure 2. A Process Flow Diagram of both the UF and the Coalescing Apparatus.

Operating Procedure of the UF Unit

1. The feed reservoir was filled with feed emulsion from the sonicator.
2. The outlet line ball valve was fully opened.
3. The pump was started and its speed was adjusted until the pressure gauge at the inlet of the UF module read 10 psi.
4. The ball valve in the outlet line was slowly closed until the pressure gauge at the UF outlet read 15 psi and the pressure gauge at the UF inlet read 25 psi, giving an average transmembrane ΔP of 20 psi. This also gave a ΔP through the hollow fibers of 10 psi, which gave sufficient hollow fiber velocity to prevent the inside fiber surfaces from fouling.
5. Steady state was reached at about 15 minutes and the following experimental data were obtained:
 - i. A 1 L beaker was inserted underneath the filtrate outlet hose barb and the volume of filtrate collected over 1 minute was recorded. The measured flow rate was 300 mL/minute.

- ii. The circulation rate on the tubeside of the unit was measured by removing the discharge line from the feed container and inserting it into a 1 L beaker and recording the time required to circulate 1 L of feed. The experimental data showed that 0.5 gpm was recirculated.
6. After 15 minutes the volume of the 5 L feed tank was decreased to 0.5 L at which time the level in the feed tank was too low to provide suction for the pump, and at which time the contents of the UF feed tank were pumped to the graviy separator of the coalescing unit.
7. The operation of the UF unit for one batch cycle is summarized as follows:
 - i. The filtrate rate was 0.3 L/min.
 - ii. The circulating rate on the tubeside was 0.5 gpm.
 - iii. 15 minutes were required to reduce the UF feed from 5.0 to 0.5 L.
 - iv. 0.5 L of concentrated oil/water emulsion was transferred to the coalescing unit gravity separator.

Ultrafiltration Unit Operation

A 5 L jug was used as the feed container to supply the sonicated oil-water emulsion. The emulsion was pumped from the container using the 1 gpm Procon pump and fed to the Romicon UF module. The retentate of the UF unit was continuously recycled to the feed container while the filtrate was collected in a beaker. The pump speed and the ball valve were manipulated to obtain a transmembrane pressure of 20 psi and a module outlet pressure of 15 psi.

Coalescer Unit

Equipment

1. Coalescing Chamber

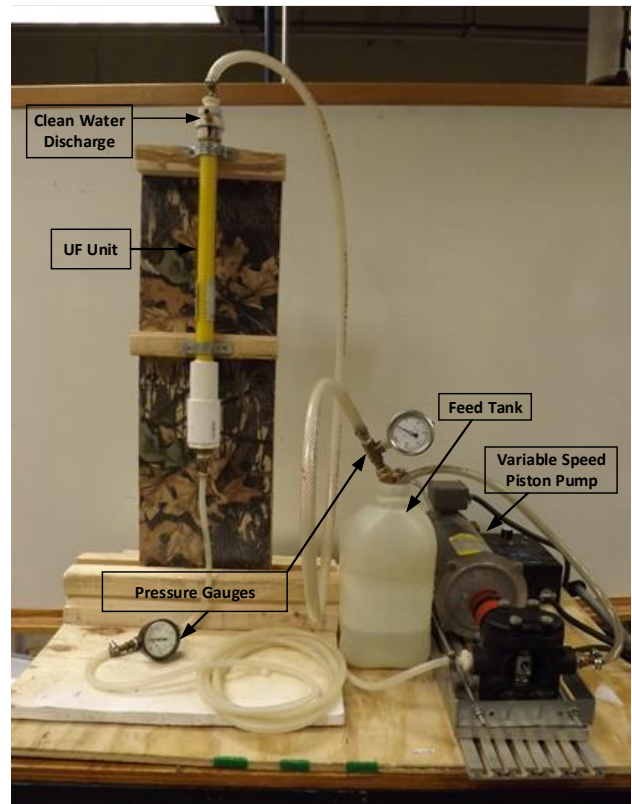


Figure 3. A Diagram of a UF System

- a. A 1" SS bolt 1" long drilled through with a 27/64" (0.422") drill.
- b. A 1" SS nut with a 1/4" thick SS disk welded onto one side; the disk was tapped for 1/4" pipe threads in its center.
- c. Eight 1" diameter disks cut from 10 micron polypropylene felt filter bags.
 - i. The measured fiber diameter was 18.3 microns.

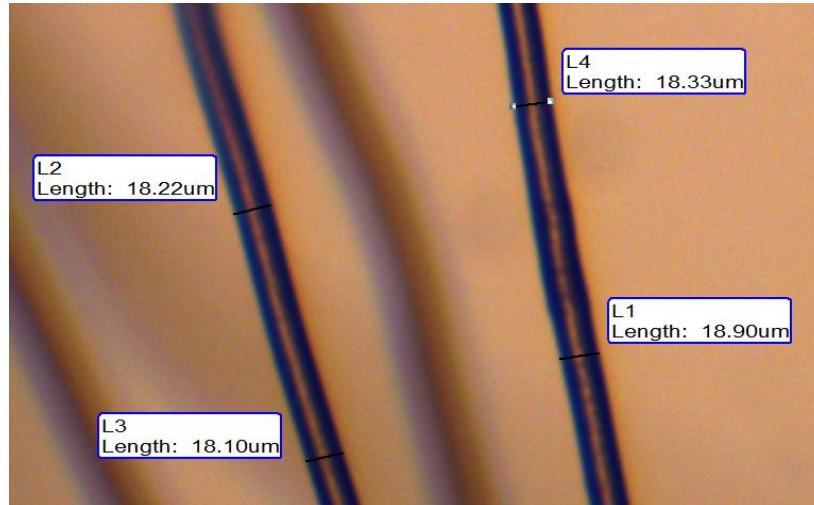


Figure 4. Microscopic view of the polypropylene fibers.

2. A gravity separator consisting of the following:
 - a. A 4" ID x 4 1/2" OD x 12.25" long acrylic tube with a 1/2" bottom plate.
 - b. The top chamber was constructed from a 4" OD (at the top) glass funnel.
 - c. The inverted glass funnel was mounted to the top of the chamber with a 20 mL plastic syringe housing (3/4" ID x 4" length) siliconed to its stem.
3. A Procon Gear Pump, Model Number 1112A060F11CA, 1 gpm at 1750 rpm.
4. A variable speed (0 to 1750 rpm) electric drive.

Operating Procedure

1. The gravity separator chamber was filled with concentrated oil/water emulsion from the UF retentate stream.
2. The pump was started and the flow through the coalescing element was adjusted to 2 L/min (0.5 gpm).
3. Additional feed was added to the gravity separator to bring the level in the 20 mL syringe housing to near its top.

4. The unit was allowed to circulate for 180 minutes (3 hours) at which time the majority of oil was separated and had either coated the inside of the gravity separator vessel or had collected in the top of the syringe housing.

5. The run was then stopped and the oil was pipetted out and the water phase was sampled.

NOTE: The clarified water phase was not recycled back to the UF unit feed in the laboratory; however, this will be implemented in the plant unit.

The coalescing chamber was constructed to the specifications above. Figure 5 shows two photographs of the coalescing unit. The polypropylene layers were cut very carefully using a milling machine with a hole punch attachment. The nut was made water tight with a liberal wrapping of Teflon pipe tape. The bolt was tightened by hand until it was certain that no channeling was occurring around the coalescing medium.

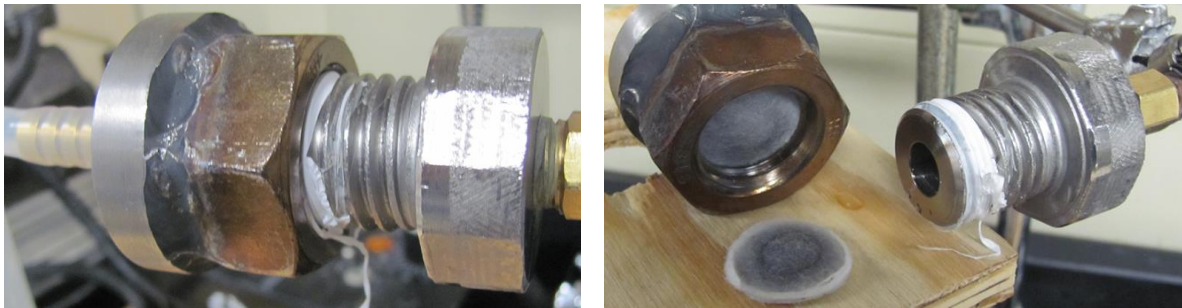


Figure 5. (Left) A close-up photograph of the assembled coalescing unit. (Right) An exploded (i.e., disassembled) photographic view of the coalescing unit.

The outlet of the coalescing chamber was fed to the top of the acrylic gravity separation tank. The vessel had an inlet with a septum through which a syringe could be used to collect samples. An outlet located at the bottom of the separation vessel was used to recirculate the emulsion through the coalescer using the pump described above. The graduated syringe was used to measure the volume of the oil layer.

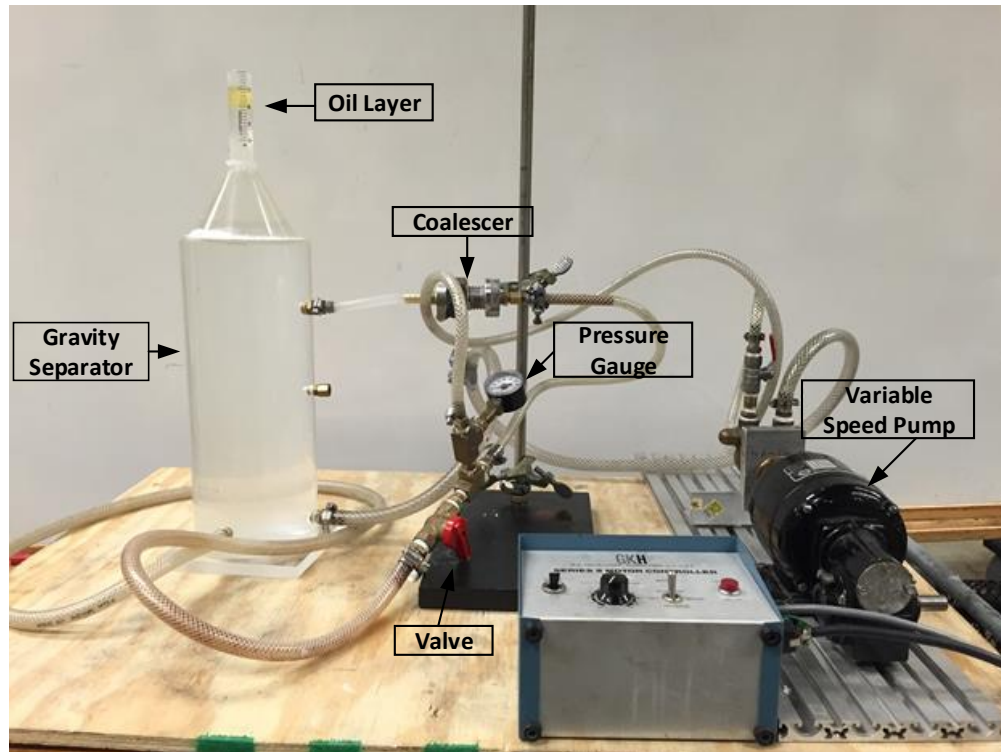


Figure 6. A photograph of the coalescing apparatus.

EXPERIMENTAL RESULTS

Experiments were conducted to verify the production of a homogeneous oil-in-brackish water emulsion and to measure the efficacy of each of the separation steps utilized by this design.

Sonication

In order to generate stable emulsions in a minimal amount of time, several experiments were performed with the sonicator. Analysis of the dispersions was conducted using a Biological Microscope XSG Series and *AMScope MT500* software. An inverse correlation between sonication time and droplet size was observed. A sonication time of 60 minutes reaches equilibrium with a number average droplet size of < 6 microns. Figures 7 through 9 show microscopic images of the produced emulsions.

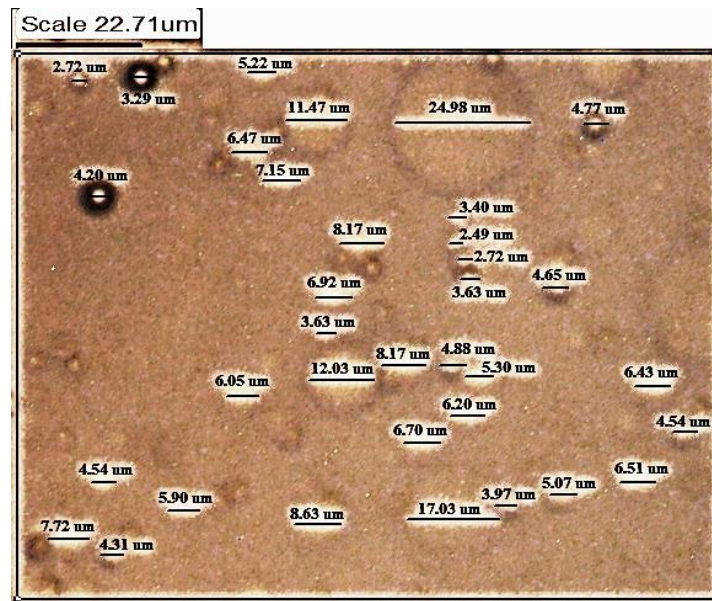


Figure 7. A sample of oil-water emulsion after 15 minutes of sonication

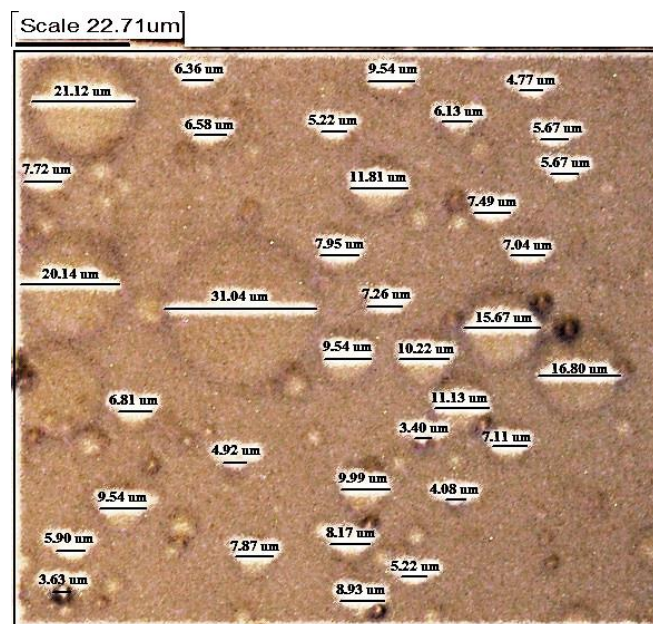


Figure 8. A sample of oil-water emulsion after 60 minutes of sonication

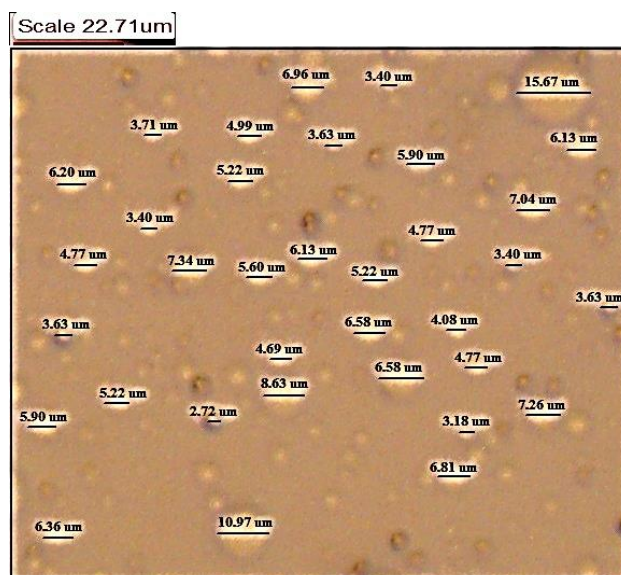


Figure 9. A sample of oil-water emulsion after 120 minutes of sonication

Oil/Water Separation

To test the performance of UF membranes, feed emulsion at a concentration of 0.22 mL/L was circulated through the apparatus until the volume in the feed tank dropped from 2,900 mL to 900 mL for a volume reduction of almost 70%. The transmittance of the filtrate was analyzed using a spectrophotometer blanked with pure un-sonicated brine. With a 98.1% transmittance, the brine recovered from the UF membrane was virtually free of any dispersed oil, leaving the retentate emulsion in the feed tank at an oil concentration of 0.70 mL/L. The comparison in the clarity of the filtrate and the untreated sonicated emulsion may be observed in Figure 10. Based on these results and a consultation with Prof. Robert Cross, a recognized UF expert, it was determined that a volume of 10 gallons could be easily reduced by over 90%, thus leaving the retentate at oil concentrations more suitable for the coalescer.¹¹

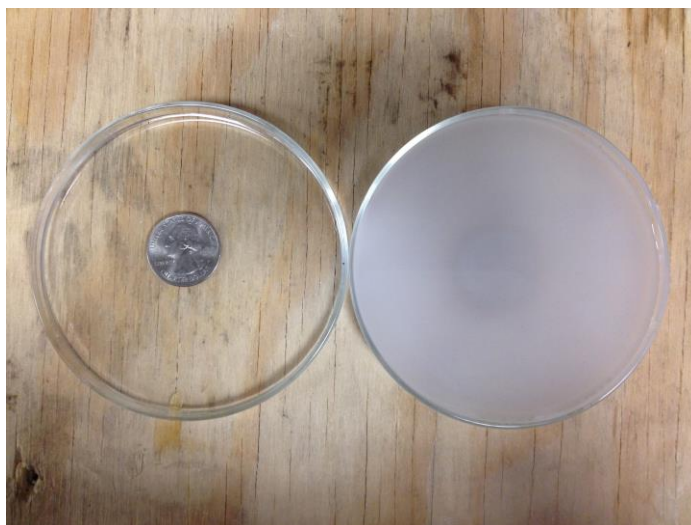


Figure 10. A Photograph of the UF filtrate (left) compared to the feed emulsion (right).

The coalescing apparatus was tested using a 2.0 mL/L oil in brackish water emulsion pumped through the coalescer at a flow rate of ~ 2.2 L/min. Figure 12 shows a plot of the transmittance and the oil accumulation for a 10 hour coalescer operating period. In under 3 hours, the coalescer was able to remove two thirds of the oil present in the solution and achieved a transmittance of over 50% relative to the brine blank. Comparatively, the concentrated emulsion that was fed to the coalescing apparatus had a transmittance of $< 5\%$. A progression of the accumulated oil layer may be observed in Figure 11 for 43, 63, 164 and 300 minutes.

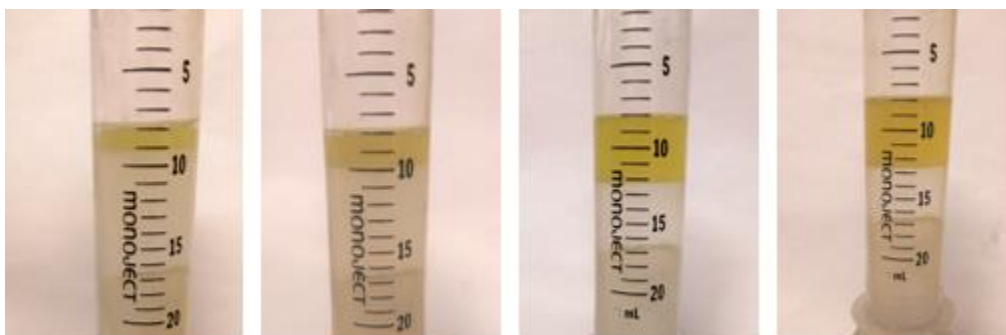


Figure 11. Photographs of the oil-phase layer taken at (from left to right) 43, 63, 164 and 300 minutes.

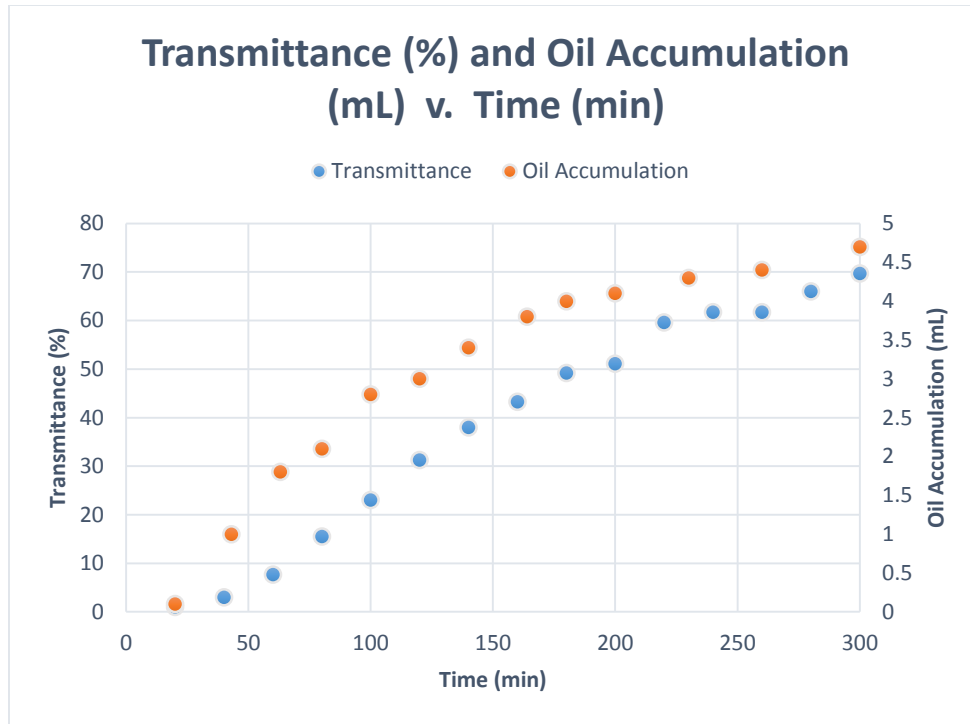


Figure 12. Plot of the transmittance (%) and oil accumulation (mL) in the coalescer for a period of 300 minutes (5 hours).

COMMERCIAL DESIGN

The equipment for the full scale system will consist of (1) 100 gpm, 3 HP centrifugal pump, (1) single housing feed filter unit, (2) 3,000 gallon, HDPE feed tanks, (6) Koch TARGA PM100 UF modules, (3) 200 gpm, 5 HP circulating pumps, (8) #1 10 micron PP filter bags, (2) 2,000 gallon, HDPE gravity separation tanks, and (1) 7.9 gpm, 1/8 HP rotary vane pump. The full scale process flow diagram is shown in Figure 13.

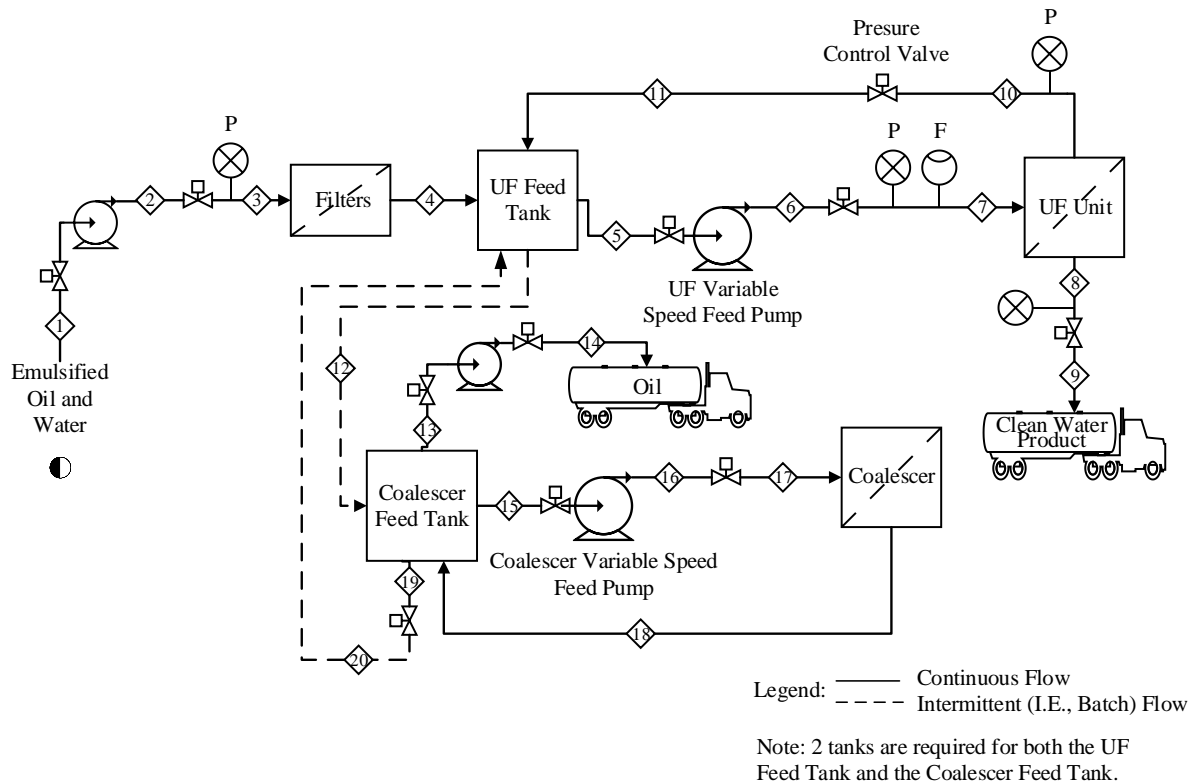


Figure 13. Process Flow Diagram for the Commercial Plant.

Scaleup

UF Modules

At a transmembrane pressure of 20 psi, the 1 ft² laboratory UF module produced 0.3 L/min of permeate. The scaled-up flow rate of 100 gpm will require a membrane surface area of 1,570 ft² (at the same transmembrane pressure). Each of the Koch TARGA PM100 UF modules has a membrane surface area of 367 ft²; therefore, five modules (with a membrane surface area of 1835 ft²) are required. A sixth module will be purchased as an installed spare.

UF Recirculation Rate

The recirculation rate of the bench scale UF unit is about 1 gpm. The laboratory unit has a bundle diameter of about 1" and a length of 18", whereas the plant unit will have a bundle diameter of 8.4" and a length of 72". Based on manufacturer's recommendations a recirculation rate of 400 gpm will give 5 psi pressure drop through the tubes of the module.

Coalescer

The flow rate though the bench scale coalescer is 0.5 gpm through slightly less than 1" diameter, which gives a specific flux of 0.67 gpm/in². At this flow rate the turnover time for the

bench scale gravity separator in the lab is about 1 minute. With a cycle time of 3 hours (180 minutes) the bench scale tank is turned over 180 times. The plant unit will be turned over about 20 times which will required a flow rate through the coalescing elements of 200 gpm. At a flow rate of 200 gpm and a required specific flow rate of 0.67 gpm/in^2 , the required coalesce area is 300 in^2 .

Oil Discharge Pump

At two volume percent oil in the feed, the oil production rate is 2 gpm. This oil will collect in the gravity separation tank and must be discharged periodically.

Process Description

The UF feed pump will take suction from a rubber lined storage pond at a fracing site. The feed pump will be a 3 HP Dayton Thermoplastic pump capable of pumping 100 gpm at 50' of head. This pump is self priming and has a housing to retain water. A hand valve and rotometer will be placed in the discharge line between the pump and the inlet filter.

The inlet filter will consist of three # 2 polypropylene bag filters in a single housing. Each bag has a filter area of 4.7 ft^2 and can handle up to 88 gpm. The dimensions of each bag filter will be 7" in diameter and 32" in length.

Two 3,000 gallon tanks will be used to store feed for the UF unit. At a 100 gpm feed rate each tank will have a 30 minute cycle time. While one tank is being filled, the other tank will serve as a batch feed tank providing feed and recirculation flow to the UF modules.

After a tank has been filled with both fresh feed and water recycle from the coalescing gravity separator it will be recirculated at a rate of 400 gpm. Recirculation will continue with 100 gpm of filtrate production until the volume in the tank is reduced to 300 gallons at which time the tank contents will be transferred to the coalescer feed tanks.

The UF recirculation pump will take suction from the UF feed tank and recirculate it through the UF modules. A manual valve on the effluent side of the UF module shell will be used to maintain an outlet pressure of 30 psi. A variable speed motor on the pumps will be used to maintain an inlet pressure of 25 psi to the recirculation stream. For the recirculation, two 5 HP Dayton Pool Pumps capable of pumping 200 gpm at 50' of head will be used.

The coalescer will consist of eight filter housings in series. Each of these will house a # 1 filter bag with 2.7 ft^2 (388 in^2) of surface area. The filter housings in series will the coalescing capability of the 8 layer filter bed used in the bench scale unit.

Two 2,000 gallon HDPE tanks will serve as the gravity separation tanks. The dimensions of the tanks will be 96” in diameter and 83” in height. On the average these tanks will take 10 gpm of feed from the UF feed tanks and have a cycle time of 200 minutes.

Once the oil layer has been established, it will be pumped out of the separation tanks and into a tank wagon. It is reasonable to allow the oil to collect to about the 200 gallon level in the 2,000 gallon gravity separation tank before discharging it. The design pumpout rate of 7.9 gpm gives a discharge rate of 25 minutes.

The entire apparatus will be mounted onto a flatbed trailer, giving the process mobile capabilities. The selected trailer is a Fontaine trailer with dimensions of 53’ in length and 102” wide.

ECONOMICS

Table 2. Economic breakdown of full scale process.

EQUIPMENT	DESCRIPTION	SPECS	PURCHASE COST
UF Feed Pumps	100 gpm Pool Pump	3 HP, 100 gpm at 50’	\$1,400
Feed Filter	(3) # 2 Bags	Single housing	\$5,000
UF Feed Tanks	(2) HDPE Tanks	3,000 gal, 102’D x 93’H	\$8,000
UF Module	(6) Koch TARGA PM100 Module	367 ft ² , 45 psi max	\$9,000
UF Circulation Pump	(2) 200 gpm Pool Pump	5 hp, 205 gpm at 50’ head	\$4,000
Coalescer	(8) #1 10 micron Bags	PP 8 in series	\$15,000
Coalescer Gravity Separators	(2) HDPE Tanks	2,000 gal, 96’D x 83’H	\$6,000
Coalescer Circulation Pump	(1) 200 gpm Pool Pump	5 hp, 205 gpm at 50’ head	\$2,000
Oil Discharge Pump	Rotary Vane Pump	7.9 gpm Positive Displacement	\$1,000
Total Purchase Cost			\$51,400
Total Installed Cost	(trailer not included)	\$51,400 x 4 =	\$206,000
Total Project Cost		\$206,000 + \$40,000 =	\$246,000

It is assumed that the operating costs are less than \$20,000/year, excluding any additional operating labor. On a 5 year payout basis the yearly capital charge is \$50,000/year; thus, excluding labor the yearly charges are about \$70,000/year. If the unit operated 8,000 hours per year, the yearly clarified brine production will be 53 million gal/year (53,000 kgal/year) giving a

cost of \$1.25/kgal. If around the clock labor is added with 4 operators at \$50,000/year per operator, then the total cost will be \$5/kgal.

WASTE DISPOSAL

Depending on the particulates in the feed, the on-stream time for a set of filter bags will vary. When the pressure drop through the bags exceeds the manufacturer's recommended limit the bags will be replaced and will be disposed of properly. The oil contamination will likely require disposal by a licensed hazardous waste company.

REGULATIONS

Safety

OSHA regulations dictate that a number of safety considerations must be accounted for in order to promote worker safety. Because this system is operated manually, the most important safety measure is to have properly trained employees that are familiar with the process. The separated oil must be handled according to state and federal regulations regarding the handling and transportation of crude oil. The process will most likely operate at a flowback water treatment site, in which case the workers must be trained to operate according to the safety guidelines applicable to the drilling site.

Employees must be informed of the various levels of hazards associated with flammable compounds, high pressure from the flowback fluid, and potentially high levels of hydrogen sulfide. One of the major considerations directly related to the separation process is the composition of the fluid. The fluid will likely contain hazardous chemical residues and elevated levels of hydrogen sulfide. To abide by OSHA regulations, the operators are required to be trained in detecting hazardous chemicals, evaluating the work environment for potential exposure to hydrogen sulfide and other hazardous chemicals, and provide appropriate information regarding personal protective equipment (PPE).^{12,13}

Hearing protection is mandated for any person in the vicinity of the sonicator when it is in use. The sonicator produces audible frequencies up to 95 dB that can potentially cause hearing damage. Signs requiring ear protection must be present in the surrounding areas to indicate when it is in operation to protect the hearing of all employees.

Environmental

There are a number of different regulations that must be met depending on the application of the recovered brackish water. If the recovered brackish water is going to be reused in fracking applications, it must meet the following criteria:

- pH between 6 and 8
- Total hardness content of 5,000 mg/L or less (based on calcium and magnesium content of the treated brine).
- Bacteria concentration no greater than 100 colony forming units/mL (2 positive bottles utilizing API RP-38 serial dilution techniques for both sulfate reducing and acid producing bacteria (SRB and APB, respectively).
- Total suspended solids content (TSS) less than 50 mg/L.
- Oil and grease (hexane extractable organics) content less than 100 mg/L.
- Soluble sulfate (SO_4^-) content less than 600 mg/L.¹⁴

CONCLUSION

1. The Ultrasonic HogUAnauts team has determined that UF in conjunction with a coalescer is the best and most cost-efficient method for recovering oil from emulsions present in fracking flowback water. The process produces virtually oil-free brackish water and an easily removed oil phase.
2. The versatility provided by a mobile process is well suited to the transient nature of the fracking industry.
3. Because all fracking wells produce water of varying oil/water compositions, tests should be conducted to determine the volume reduction that will be necessary to achieve optimal concentration for the coalescer.
4. The estimated total capital cost of the system is \$238,000.
5. The operating cost, excluding operating labor, is less than \$20,000/year.
6. On a 5 year payout basis the yearly capital charge is \$46,000/year; thus, excluding labor the yearly charges are about \$66,000/year. If the unit operates 8,000 hours/year, the yearly clarified brine production will be 53 million gal/year (53,000 kgal/year) giving a cost of \$1.25/kgal. If around the clock labor is added with 4 operators at \$50,000/year per operator then the total cost will be $1.25 + 3.8 = \$5/\text{kgal}$.

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Audits

Audit from Prof. Bob Cross of the Ralph E. Martin Department of Chemical Engineering at the University of Arkansas received March 11, 2015

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Page 4. Hydraulic fracturing, “fracking”

Page 10. Section 5 ii I assume you mean that the rate exiting the tubeside is 0.5 gpm. The rate entering the tubeside would be 0.5 gpm plus 0.3 L/min.

Page 10. Section 7 ii I think you mean gpm but see above.

Page 18. There is no need for a pump on the outlet line of the UF unit. It will just make control difficult.

Page 25. There are a number of references to the treatment of oily wastes using UF. I suggest you include a couple of them.

Audit from Alex Lopez of the Ralph E. Martin Department of Chemical Engineering at the University of Arkansas Graduate Program received March 13, 2015

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Page 3. Italicize “in situ”

Page 3. Last paragraph, what is in the feed?

Page 4. Introduction paragraph, what is flowback water?

Page 4. Where is 1st citation?

Page 5. Task Parameter, You have periods on some but not all. Pick one.

Page 5 & 6. References?

Page 7. Italicize “a priori”

Page 12. Operating Procedure, How can this be continuous if oil coats the instrument?

Page 17. Figure 10, which is which?

Page 18. Figure 11, Use different shapes!

Page 24. Can this be profitable?

Audit from Byron Hinderer 50 plus years of experience in the engineering field received March 13, 2015

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Phone: (512) 258-3484

Page 4. Make the sequence and phrasing of the technologies introduced match those in the technologies considered.

Page 5. It is essential to describe each process thoroughly so that the generalized descriptions throughout the rest of the document can be eliminated.

Page 6. State advantages and disadvantages for each technology considered.

Page 7. If testing was done on technologies considered, should mention methodology used, objective and goals, statement of results, and a conclusion.

Page. 7. Make all the information consistent with the summary and conclusions.